COLLODION MEMBRANES OF HIGH PERMEABILITY

COLUMBIA UNIVERSITY

DISSERTATION

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN THE FACULTY OF PURE SCIENCE, COLUMBIA UNIVERSITY

BY

DAVID PERCY MORGAN, JR.

NEW YORK CITY
1923

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COLLODION MEMBRANES OF HIGH PERMEABILITY.

Various methods of preparing collodion membranes of graded permeability have been previously described by Bechhold (1), Walpole (2), Brown (3), and Eggerth (4). These several investigators have arrived empirically at different procedures for producing essentially the same result. When, however, a reproducible graded membrane of high degree of permeability is desired, none of their methods is entirely satisfactory.

In the work described in this paper, flat membranes, prepared according to Walpole, were used. By Walpole's procedure collodion solution is poured upon a carefully levelled circular glass plate in sufficient quantity to spread evenly to the edges of the plate. As the alcohol-ether solvent evaporates the solution becomes more and more viscous until finally a membrane is formed. The plate, with its film, is now immersed in water and after the lapse of a few minutes the membrane can be easily removed.

Walpole observed that by water immersion of the plate and film at different stages in the evaporation of the solvent, a series of membranes of different degrees of permeability could be obtained, since in this way the amount of residual solvent to be replaced more or less by water is varied. This relationship between the amount of water and solvent finally contained in the film, which Walpole has termed the "wetness," and the degree of permeability of the film has since been substantiated by Brown (3) and Eggerth (4).

In the present work the "wetness" per gram of dry collodion is termed the "grade" of the membrane and is determined directly

by weighing. This procedure constitutes an improvement over Walpole's measurement of the thickness, since it permits determination of the grade of a membrane at any point in the process without waiting until the membrane becomes tough enough to measure with a caliper. A further advantage is the possibility of working with very much thinner membranes (about 0.01 mm. versus Walpole's 0.1 mm.).

In working with these thin membranes the very interesting observation was made that the change from viscous liquid to rigid jelly is quite abrupt. It thus becomes possible to detect and measure an important property of the membrane; namely, the grade of the membrane at the instant of jellification, the so called "initial gel grade."

By means of this initial gel grade it is possible to measure the tendency of a given sol to set to a jelly, and a direct way is thereby provided for studying the conditions favorable to the formation of jellies of high degree of wetness and hence of high permeability. From the foregoing it is obvious that one very important condition favoring high permeability is the immersion of the membrane in water as quickly as possible after the point of jellification has been reached.

Relation between the Composition of the Collodion Sol and the Initial Gel Grade.

The actual method finally adopted for this work combines several features of the different techniques previously mentioned. Thus the solutions are prepared, as by Eggerth (4), by dissolving a given weight of Du Pont's parlodion in 100 gm. of absolute alcohol and ether, mixed in stated proportions by weight; and a measured volume of the given solution is then poured, as by Walpole, on a plate of known weight. If too much collodion is used the surface of the thick membrane formed gels somewhat sooner than the main portion of the liquid and there is consequently no *sharp* point of transition from sol to gel. With a sufficiently thin membrane, however, this transition is abrupt and is readily observed. If changes in the consistency of the drying film are followed by gently touching it from moment to moment with a pointed match it will be observed that somewhat before jellification the rapidly thickening material adheres

strongly to the match as the latter is withdrawn; then, in quick succession, pressure with the match produces a slight temporary depression of the gelled surface into the still liquid sublayer, and, *immediately*, afterward, a semipermanent deformation, indicative of complete rigidity or jellification throughout the entire film. At this instant the plate is placed on the balance and weighed as quickly as possible to the nearest 10 mg. This weighing gives W_o, the weight of collodion plus the organic solvent retained in the jelly. As the solvent is evaporating at the rate of about 50 mg. a minute and as it takes about half a minute to make the weighing the measurements will run low by about 25 mg. However, since this error is always in the same direction and since the weights obtained in different experiments are found to differ by hundreds of milligrams no correction was introduced for this error.

As soon as the weight is determined the plate is immersed in water and left there 15 or 20 minutes until, on raising it, the water is found to wet the membrane surface. The membrane can now be weighed, after drying between filter papers, either on the plate, or by itself after removal therefrom. This determination, W_w, is more accurate than that of W_o as the water evaporates at the slower rate of about 10 mg. per minute from the membranes used. The dry weight, D, of collodion is finally determined to about 0.5 mg. after drying overnight at 70°C. From these data, G_o, the grams of solvent per gram of collodion before water immersion, and, G_w, the grams of solvent per gram of collodion after water immersion are calculated as follows:

$$G_o = \frac{W_o - D}{D}$$

$$G_w = \frac{W_w - D}{D}$$

G_o is the "grade" of the organo-jelly, and G_w is the "grade" of the final membrane.

Using the above described procedure the first question investigated was the effect on the initial gel grade of variation in the parlodion content of solutions of constant alcohol-ether composition. Previous work by Bechhold (1) indicated clearly

the existence of such an effect. In each experiment 50:50 (by weight) alcohol-ether solutions of suitable parlodion content were used. Each membrane was numbered by inserting a small paper tag in the rim of the jelly. The results are shown in Table I.

TABLE I.

Effect of Variations in the Parlodion Content of the Sol upon the Initial Gel Grade, G_o, and the Final Grade of the Membrane, G_w.

Alcohol-ether ratio = 50:50 in each experiment.

Membrane No.	Parlodion per 100 gm. solvent.	Sol used.	Time to set.	W_o	W_w	D	G_o	G_w	R
	gm.	*cc.*	*min.*						
108	6	10	13	3.413	2.068	0.423	7.1	3.9	1.82
109	6	10	9	3.382	1.965	0.423	7.0	3.6	1.94
110	4	8.5	10	2.892	1.830	0.247	10.7	6.4	1.67
111	4	8.5	9	2.790	1.760	0.247	10.3	6.1	1.69
112	4	8.5	95	2.875	1.850	0.245	10.7	6.1	1.76
113	4	8.5	95	2.832	1.760	0.243	10.7	6.2	1.73
114	3	7.5	9	2.500	1.725	0.163	14.3	9.6	1.49
115	3	7.5	85	2.413	1.650	0.164	13.7	9.1	1.51
116	3	7.5	9	2.542	1.755	0.166	14.3	9.6	1.49
117	3	7.5	9	2.440	1.630	0.166	13.7	8.8	1.56
118	2	5	6	1.404	0.960	0.086	15.3	10.2	1.50
119	2	5	6	1.645	1.150	0.092	16.9	11.5	1.47
120	2	5	6	1.500	1.084	0.086	16.5	11.6	1.42
121	2	5	6	1.450	1.070	0.086	15.9	11.4	1.40
122	2	5	6	1.415	1.050	0.086	15.4	11.2	1.38
123	2	5	6	1.483	1.085	0.089	15.7	11.2	1.40

The first four columns give, successively, the number of the membrane, the grams of parlodion per 100 gm. of solvent, the volume in cubic centimeters of sol required to cover the plate, and the time required for the jelly to form. The next three columns give the observed weights of the membranes: W_o, the combined weight of parlodion and organic solvent at the moment the jelly sets; W_w, the weight of parlodion plus solvent after immersion; and D, the weight of the membrane after drying overnight at 70°C. From these data, G_o, the "initial gel grade,"

that is, the grams of organic solvent per gram of parlodion before immersion; and G_w, the grams of solvent per gram of parlodion after immersion, is calculated as previously described.

In the last column, headed R, is given the ratio of the grade before and after immersion in water.

$$\left(R = \frac{W_o - D}{W_w - D}\right)$$

A comparison of the values of G_o, the initial gel grade, for groups of membranes made from solutions of the same collodion content shows that the new method of determining this property gives satisfactory results. The reliability of the method seems therefore established. A further point of interest is brought out; namely, that the initial gel grade, G_o, from a 2 per cent sol is more than twice as large as G_o from a 6 per cent sol.

This would naturally suggest that further increase in grade could be attained by the use of still more dilute solutions, but experiment quickly demonstrated that the resulting films are too fragile to be used. The remaining experiments in this paper have therefore been carried out with 2 per cent solutions.

Having established an optimum parlodion concentration we next studied the effect of varying the alcohol-ether ratio in the solvent mixture. The existence of such an effect was indicated clearly by the work of Eggerth (4). The application of the initial gel grade method to this problem yielded the results shown in Table II.

The results in the G_o column agree in general with the work of Eggerth in showing that the grade increases with alcohol content of the solvent. Not only, however, has the present method yielded membranes of a grade more than twice as high as any reported by Eggerth (compare our $G_w = 17$ with Eggerth's highest grade of $G_w = 8.43$); even more to be emphasized is the elimination by the present technique of the uncertainty involved in the employment of any drainage method. When films are produced by dipping and drainage, as in Eggerth's work, the properties of the membranes produced depend upon the simultaneous influence of at least three factors: (1) the viscosity of the solution which affects the rate of drainage and consequently the thickness of the film at various levels; (2) the rate of evaporation,

which affects, on the one hand, the drainage, and, on the other, the rate of gel formation; and (3) the critical concentration at which jellification occurs. The initial gel grade method eliminates drainage as a factor and likewise by referring all observations to a standard condition of the film, regardless of the time consumed in reaching that condition, eliminates rate of evaporation, leaving the critical concentration as the only variable. The favorable effect of increased alcohol concentration on the grade of the membrane can therefore now be definitely attributed to an influence on the sol-gel transformation.

TABLE II.

Effects of Variations in the Ratio of Alcohol to Ether in the Solvent.
5 cc. of 2 per cent parlodion sol were used in each experiment.

Alcohol-ether ratio by weight.	Membrane No.	W_o	W_w	D	G_o	G_w	R
25:75	124	0.904	0.665	0.083	9.9	7.0	1.42
	125	0.876	0.640	0.085	9.3	6.5	1.43
	126	0.980	0.690	0.084	10.6	7.2	1.47
	127	0.933	0.645	0.085	10.0	6.6	1.52
50:50	118	1.404	0.960	0.086	15.3	10.2	1.50
	119	1.645	1.150	0.092	16.9	11.5	1.47
	120	1.500	1.084	0.086	16.5	11.6	1.42
	121	1.450	1.070	0.086	15.9	11.4	1.40
75:25	128	1.912	*	0.081†	21.9		
	129	2.101	1.480*	0.082	24.6	17.1	1.44
	130	2.082	1.410*	0.079	25.4	16.9	1.5
	131	2.045	1.380*	0.082	23.9	15.8	1.51

* Damaged in removal from plate.
† 0.081 is the average D for Membranes 129, 130, and 131.

The practical employment of our most permeable membranes in a study of the diffusibility of invertase was prevented, however, by the impossibility of removing them intact from the plate. Fortunately, a very simple way of overcoming this difficulty was soon found, for it is only necessary to fog the glass surface before pouring on the collodion sol in order to obviate the tendency to stick. This is readily accomplished by breathing on the plate which, if necessary, has been chilled to a few degrees below room

temperature. A light fog is all that is necessary; a heavy deposit of moisture is to be avoided, as the membrane may be damaged. Fogging also provides a test for the cleanness of the surface, for, if an invisible film of collodion or oil is present, the fog will not form evenly.

It is of interest to note that even the small amounts of moisture introduced into the membrane by this fogging process have a well defined effect on the initial gel grade. To illustrate this point Table III is introduced. In each experiment of this series 5 cc.

TABLE III.

Effect of Fogging the Plates.

5 cc. of 2 per cent parlodion sol of alcohol-ether ratio 75:25 were used in each experiment.

	Membrane No.	W_o	W_w	D	G_o	G_w	R
Plates not fogged.	128*	1.912		0.081†	21.9		
	129*	2.101	1.480	0.082	24.6	17.1	1.44
	130*	2.082	1.410	0.079	25.4	16.9	1.5
	131*	2.045	1.380	0.082	23.9	15.8	1.51
Plates fogged.	136‡	2.273	1.565	0.079	27.8	18.8	1.48
	137‡	2.286	1.555	0.080	27.6	18.5	1.49
	138‡	2.282	1.580	0.081	27.2	18.5	1.47
	139‡	2.264	1.540	0.080	27.3	18.3	1.49
Plate not fogged; 2 per cent H_2O in sol.	146*	2.602	2.086	0.080	31.5	25.1	1.26
	147*	2.534	2.000	0.080	30.7	24.0	1.28
	148*	2.612		0.080†	31.6		
	149*	2.546		0.080†	30.9		

* Damaged in removal from plate.

† Average D for Membranes 129, 130, and 131.

‡ Came off plate easily.

of a 2 per cent parlodion, 75:25 (by weight) alcohol-ether sol, were used. The membranes were immersed in water as soon as the initial gel weight was determined. The effect of moisture on the initial gel grade, G_o, is evident from comparison of Experiments 128 to 131, where no moisture was deliberately added, with Nos. 136 to 139, where moisture was added by fogging the plate, and with Nos. 146 to 149, where moisture was added directly to the solution.

These results suggested a new way of increasing the grade of the membranes, but unfortunately, very little advantage could be gained by adding water to the sols because even small amounts of water have the undesired effect of making the membranes too fragile for use. The practical value of these observations consists in the parallel increase in initial gel grade and fragility, thus suggesting precautions to be adopted in order to obtain films of high grade, and at the same time reasonable strength. Aside from the obvious precautions taken for the dryness of the solvent alcohol-ether, there are still two ways by which moisture may get into the membrane. First, the worker may inadvertently breathe on the solution on the plate while watching for the initial gel

TABLE IV.

Effect of Humidity on the Initial Gel Grade, G_o.

In each experiment the plate was fogged and 5 cc. of a 2 per cent parlodion sol of alcohol-ether ratio (by weight) equal to 75:25 were poured upon it.

	Membrane No.	W_o	D	G_o
Relative humidity less than 50 per cent.	171	1.904	0.076	24
	172	1.934	0.076	24.4
Relative humidity; sol breathed on.	173	2.346	0.076	29.9
	174	2.278	0.076	29.0
Relative humidity about 80 per cent.	176	2.213	0.076	28.1
	177	2.338	0.076	29.7
	178	2.342	0.076	29.8
	179	2.309	0.076	29.4

point; and second, if the relative humidity be sufficiently high, moisture may be taken up from the atmosphere to the detriment of the membrane. Cases illustrating these effects are shown in Table IV, which is self-explanatory.

We suggest as a plausible, though incomplete, explanation of the rôle of water in these experiments that moisture in the sol causes a precipitation of the collodion concomitantly with gel formation. This tentative explanation seems justified by the observation that the fragile jellies are more opalescent than the stronger ones and that relatively slight further increases in water content cause distinct precipitation in separate white flocks.

It will be noted from our tables that the ratio G_o:G_w is always greater than unity. This indicates a loss of permeability during the immersion of the membrane in water. It was thought that if the replacement of organic solvent by water were allowed to take place more gradually these losses might be diminished. An increase in the grade after water immersion was actually accomplished by dipping successively in alcohol-water solutions of decreasing alcohol strength. The results are shown in Table V.

TABLE V.

Effect on Final Grade, G_w, of Immersing the Organo-Gel in Alcohol-Water Solutions of Successively Lower Alcohol Strengths.

5 cc. of 2 per cent parlodion sol of alcohol-ether ratio = 75:25 were used in each experiment.

	Membrane No.	W_o	W_w	D	G_o	G_w	R
Plate fogged; direct water immersion.	136	2.273	1.565	0.079	27.8	18.8	1.48
	137	2.286	1.555	0.080	27.6	18.5	1.49
	138	2.282	1.580	0.081	27.2	18.5	1.47
	139	2.264	1.540	0.080	27.3	18.3	1.49
Plate fogged; graded alcohol immersion.	161	2.210	1.800	0.076*	28.1	22.7	1.24
	162	2.150	1.730	0.076	27.3	21.8	1.25
	164	2.230	1.840	0.076	28.3	23.2	1.22
	165	2.186	1.875	0.076	27.8	23.6	1.13

* 0.076 is the average D for three other membranes prepared in the same way.

In the last five experiments (Nos. 161 to 165), the membranes were immersed successively in alcohol-water solutions of the following strengths, by volume, of 95 per cent alcohol: 80, 70, 60, 50, 40, 30, 20, 10, and 0 per cent. A membrane is kept in each succeeding solution from 2 to 10 minutes until on raising it the surface is wetted by the solution.

It is to be noted first, that the initial gel grade, G_o, values agree very well throughout both groups of experiments and second, that the final grade, G_w, shows a considerable increase from an average value of about 18.5 (in the first series) to an average value, in the graded alcohol experiments, of about 23.1. The column headed R shows the marked change in G_o:G_w from the usual value of about 1.5 to a lower value of about 1.2. These mem-

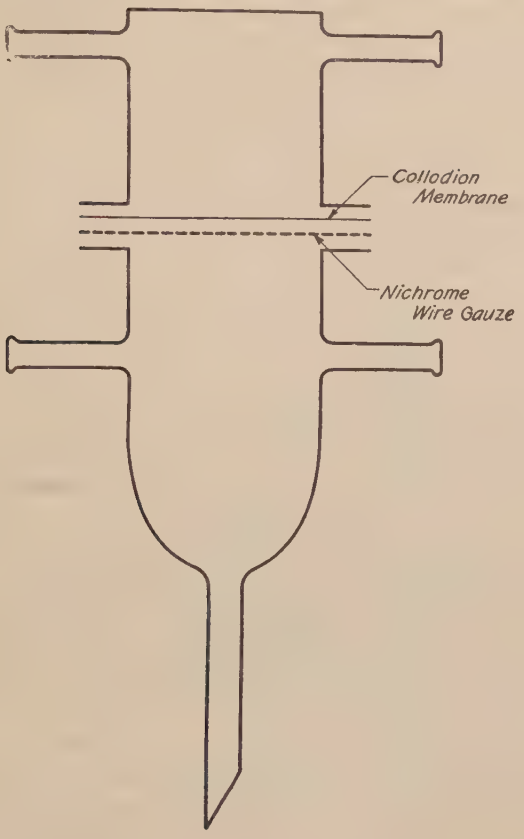
branes, and others of the same grade, were easily removed from their plates and were actually used in experiments on the diffusibility of invertase.

An additional point of practical importance is the possibility of preparing by our method in a simple manner a membrane of any desired permeability, by allowing one of a higher grade to evaporate on the balance till the desired weight is reached. Thus to make a series of membranes of G_w equal to 23 it is necessary only to make a number of membranes whose G_w is higher than this and evaporate them down to the desired grade.

A final difficulty which had to be overcome in order to render our membrane suitable for the study of invertase arises from the unavoidable variations in permeability at different parts of the film. This phenomenon was observed by Bechhold (5) when he forced air under known pressure through the membrane into a thin layer of supernatant water. Certain points in the surface allow the passage of air at pressures insufficient to force air generally through the film. The precaution was therefore taken of using a mat of three membranes in each experiment reported below with the hope of reducing the chance of one part of the surface being very much more permeable than another.

Procedure for Membrane Preparation.—As a result of the previous experiments, the following procedure for preparing membranes for ordinary use has been developed. The outside diameter of the flanged tubes of the filtration apparatus to be described later was 3 inches, and, therefore, 4 inch circular glass plates were used for the preparation of the membranes. One of these plates (the plates were always cleaned before using with cleaning mixture—concentrated sulfuric acid, saturated with potassium dichromate—washed, rinsed with dust-free distilled water, and dried in the oven at 50°C.), chilled if necessary, is weighed, carefully levelled on the desk, fogged, and 5 cc. of a 2 per cent solution of Du Pont's parlodion in 75:25 (by weight) alcohol-ether solvent mixture are run upon it from an ordinary pipette and allowed to evaporate. It is a good idea to guard the solution, as it evaporates, from convection currents and draughts by a cylindrical shield which also slows the process, making evaporation more even, and decreases the chance of moisture from the atmosphere spoiling the membrane. As soon as a jelly

forms, the plate is removed to a balance and evaporation is allowed to proceed down to a weight which has been found by a trial or two to give a grade after water immersion (G_w) somewhat higher than that desired. The immersion may be in water directly, or



FILTRATION APPARATUS

FIG. 1.

this may preferably be preceded by immersion in graded alcohol-water mixtures. The water-wet membrane is worked loose from the plate under water by gentle lateral pressure of the finger tips, and set aside to soak in water overnight. This soaking is advised because it is occasionally found that a membrane which has

possibly been hurried through the solvent replacement process will lose weight during the first few hours. After this alteration has taken place, the membranes will keep their grades for weeks. At the same time that the soaking process is taking place, a few membranes are dried in the oven to get the dry weight (D), which must be known to calculate the W_w corresponding to a certain desired G_w or *vice versa*. When a series of membranes of variable grades but all greater than the one desired is ready, it is only necessary to evaporate each on the balance to the predetermined weight W_w. The only important error in G_w is in the assumption that all membranes made from the same solution will have the same dry weight, but on the average this error is found to be less than 2 per cent. For example, the dry weights (D) of four samples from one series of membranes were in grams, 0.077, 0.076, 0.080, and 0.077, where the average deviation from the mean is 1.3 per cent.

Apparatus.—For the convenient use of these membranes in ordinary work a special apparatus was necessary. In Fig. 1, one form of this apparatus is shown which was designed and used in filtration experiments. Except for the differences in the inlet and outlet tubes the two parts are essentially the same. They consist simply of two pieces of heavy walled tubing 2 inches in diameter which are flanged and ground to fit each other. The convenient feature is the method of holding the two flanges tightly together when the membrane is placed between. This is accomplished very simply and effectively by snapping a number of strong rubber bands over the arms.

In experiments where it was necessary to brace the membranes this was satisfactorily accomplished by the use of an ordinary nichrome gauze. Such a gauze is sufficiently stiff, yet has a comparatively open mesh. In order to make this system water- and air-tight the meshes of the gauze where they come in contact with the flanges, are filled with a special gummy preparation consisting of rubber dispersed in paraffin. (This and several other valuable suggestions were given to the authors by Dr. T. C. Taylor of this University.)

In the filtration experiments about to be described this apparatus was set up in a suction flask which was connected to a mercury or water manometer and an ordinary suction pump.

This relatively simple arrangement is possible because only low pressures are needed due to the great permeability of the membranes and the low osmotic pressures developed by the colloidal solutions investigated. (Compare Bechhold (1) and McBain and Jenkins (6).)

TABLE VI.

Filtration Experiment F_3-V-A, Using Invertase Solution "W. E. M." at pH 7.5, External Pressure (about 0.03 Atmosphere) and Time (11 Hours) Same as in Experiment F_3-V-B.

Membranes { 156 (G_w = 21.8), 158 (G_w = 22.5), 159 (G_w = 22.1) }

	$V_{O,R,F}$	$K_{O,R,F}$	$K_x = K_o$ reduced to V_R or V_F.	Distribution of invertase.
	cc.			*per cent*
Original invertase solution, O	60	0.00330		
Residual solution, R	22	0.00383	0.0090	42.6
Filtrate, F	36	0.00243	0.0055	44.2
Total accounted for				86.8
Lost (by difference), *per cent*				13.2

TABLE VII.

Filtration Experiment F_3-V-B, Using Invertase Solution "W.E.M." at pH 7.5, External Pressure (about 0.03 Atmosphere) and Time (11 Hours) Same as in Experiment F_3-V-A.

Membranes { 163 (G_w = 23.2), 164 (G_w = 23.6), 165 (G_w = 23.3) }

	$V_{O,R,F}$	$K_{O,R,F}$	$K_x = K_o$ reduced to V_R or V_F.	Distribution of invertase.
	cc.			*per cent*
Original invertase solution, O	60	0.0033		
Residual solution, R	19	0.00427	0.0104	41
Filtrate, F	40	0.0022	0.00495	44.7
Total accounted for				85.7
Lost (by difference)				14.3

Filtration Experiments.

The high degree of permeability and the reproducibility necessary for a study of the diffusibility of invertase seemed now to be attained, and, therefore, we proceeded to test our membranes by actual filtration experiments. Equal volumes of the same invertase solution were placed in two of the above described vessels and subjected to ultrafiltration through equal areas of filter for equal times under identical external pressures of about 3 cc. of mercury.

The concentrations of invertase in the original solution, in the residual solution, and in the filtrate were assumed proportional to the monomolecular constant (K) of inversion of cane-sugar when equal volumes (2.0 cc.) were added to 98 cc. of 16 per cent sugar solution, buffered by citrate at pH 4.5. Comparison of the observed K with the K calculated on the assumption that all the invertase has been concentrated in the observed volume of filtrate or residual solution gives a measure of the yield of sucrolytic material present. The results of two such experiments are shown in Tables VI and VII.

The data show that the membranes are quite permeable to invertase and that two different set-ups can be made which check fairly well. The fact that the residual solutions were dark brown, while the filtrates which contained a considerable portion of the invertase were nearly water-white, points to the possibility of a purification of invertase solutions by fractional ultrafiltration, and it is hoped that the optimum conditions for the application of this method of purification can be reported in a succeeding paper.

SUMMARY.

1. A method has been devised for studying conditions requisite to the formation of collodion membranes of high permeability.

2. By means of this method the optimal concentrations of collodion and alcohol for ordinary working solutions have been roughly defined.

3. By means of an improved technique membranes have been prepared, characterized by reasonable strength, and, at the same time, a permeability almost three times as great as any previously reported.

4. A simple procedure is outlined for the preparation of a series of membranes of any desired grade, provided only that the grade desired is below the limit fixed by the fragility of the membranes.

5. Under obvious precautions the suitability of such membranes for the study of invertase diffusibility has been established.

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VITA.

David Percy Morgan, Jr. was born in Lenox, Mass., on Sept. 4, 1894. He was graduated from Middlesex School in 1912, and received the degree of A.B. at Harvard University in 1916. War service overseas as Lieutenant U. S. N. R. F. in the Northern Bombing Group interrupted studies begun at Columbia University, which were resumed in 1919 and continued, with some experience as laboratory assistant, to the present time.